

# Effect of Ytterbium Substitution on the Structural and Magnetic Properties of Nanocrystalline Zinc Ferrite

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In recent years, partial rare earth substitution for iron has been widely used to modify the composition of spinel ferrites. Analysis of published data indicates that the addition of even small amounts of rare-earth metals to ferrites changes their magnetic, electrical, and structural properties. The addition of  $\text{Dy}^{3+}$  cations to a Ni-Cu-Zn ferrite improves the magnetic properties of the material. According to Sattaret al. [6],  $\text{Yb}^{3+}$  cations increase the magnetic permeability of a Cu-Zn ferrite, whereas  $\text{La}^{3+}$  cations were reported to reduce the magnetization and coercive force of a Zn-Cu-Ni ferrite and raise the activation energy for conduction in this material. The enhancement of the magnetic and electrical properties of the spinel ferrites is commonly interpreted in terms of the magnetocrystalline anisotropy of the rare-earth elements and the exchange interaction between the  $\text{Fe}^{3+}$  and  $\text{Ln}^{3+}$  cations. At the same time, attention should be paid to the fact that the reported composition ranges of the forming solid solutions differ significantly.

Rare earth doped Zinc ferrites were prepared  $\text{Zn}(\text{Yb}_x\text{Fe}_{1-x})_2\text{O}_4$  with ( $x=0.0000, 0.0125, 0.0250, 0.0375$  and  $0.0500$ ) by the solid state method at  $900^\circ\text{C}$  to be single-phase. The cubic cell parameter  $a$  of the spinel phase was shown to increase linearly from  $8.47 \text{ \AA}$  to  $8.55 \text{ \AA}$ . However, the  $a$  cell parameter of the samples was found to increase over the entire composition range studied ( $x=0.0000, 0.0125, 0.0250, 0.0375$  and  $0.0500$ ). Note that, In the presence of impurity phases, the rate of the increase in the  $a$  cell parameter of the cubic spinel phase was slower. The formation of a limited

## ABSTRACT

A series of ytterbium substituted Zinc ferrites  $\text{Zn}(\text{Yb}_x\text{Fe}_{1-x})_2\text{O}_4$  with ( $x=0.0000, 0.0125, 0.0250, 0.0375, 0.0500$ ) was synthesized by the solid state method. The structural characterizations of all the prepared samples were done by using X-ray diffraction (XRD). These studies confirmed the formation of single-phase structure in all compositions. The increase in the value of lattice parameter with increase in ytterbium concentration suggests the expansion of unit cell. Crystallinity and the crystallite size are observed to increase with the concentration of Ytterbium. The substitution of ytterbium strongly influences the magnetic characteristics and this is confirmed from the magnetization measurements at room temperature.

**KEYWORDS:** ytterbium, XRD, FTIR, magnetic property

## I. INTRODUCTION

This paper deals with the preparation conditions, composition Ranges, structure, morphological characteristics, and magnetic properties of solid solutions based on rare-earth-doped  $\text{ZnFe}_2\text{O}_4$  zinc ferrite with the spinel structure. This ferrite is used as a material for electromagnetic radiation absorption and should have high resistivity, low dielectric losses, high hardness, high Curie temperature, and good chemical stability. There are a variety of approaches, including compositional changes, for improving the performance of microwave-absorbing zinc ferrite-based coatings in order to reduce their reflectivity for electromagnetic waves either in a wide frequency range or near one frequency of interest.

solid solution series was also reported for  $\text{YbZnFe}_2\text{O}_4$  ferrites. The  $x=0.0000, 0.0125, 0.0250, 0.0375$  and  $0.0500$  material synthesized at  $900^\circ\text{C}$  was reported to be single-phase, whereas the materials of this composition prepared at  $1100^\circ\text{C}$ , and  $1200^\circ\text{C}$  contained ytterbium ferrite,  $\text{YbFeO}_3$ .

These findings are consistent synthesis, structure, and magnetic properties with reported results for  $\text{YbZnFe}_2\text{O}_4$  ferrites, which demonstrate that the maximum degree of rare-earth substitution for Fe is  $x = 0.0500$ . In this paper, we report the preparation of zinc ferrites with the general formula  $\text{Zn}(\text{Yb}_x\text{Fe}_{1-x})_2\text{O}_4$  ( $x = 0.0000, 0.0125, 0.0250, 0.0375$  and  $0.0500$ ), doped with light and heavy lanthanides (Nd, Gd, Yb, and Lu).

The ferrites were obtained by two processes: self-propagating high-temperature synthesis (SHS) and thermal decomposition of carboxylates (salts of carboxylic acids). We determined the phase boundaries of the corresponding solid solution series and investigated the crystal structure, microstructure, and magnetic properties of ceramic samples.

## II. EXPERIMENTAL PRODUCTURE

The solid state method relies on the heat treatment of a compacted mixture of iron, zinc, and rare-earth nitrates. As starting chemicals for the preparation of mixtures, we used ZnO (analytical grade), Fe (metal),  $\text{Yb}_2\text{O}_3$  (Yb = Nd, Gd, Ln, Lu; 99.98%). An appropriate mixture of the starting chemicals was loaded into an Agate mortar and ground for 5 hours. After mixing and grinding, the mixture has been pre

sintered at 900°C for 5 hours in a furnace with heating rate of 20°C/min and cooled to room temperature with the same rate. After that, the powder and the mixture has been ground with an Agate mortar for 1 hour. Then, the powder has been pressed into pellets and toroids by uniaxial hydraulic press at a pressure of 5 tons.

The sintering temperature should start from 1000°C and 1100°C which is 100 °C higher than the pre-sintering temperature. This condition is required for densification of ferrite in standard solid state methods. Therefore, the final heat treatment step was carried out at temperatures from 1000°C to 1100°C with time duration for 5 hours to densify ZnFe<sub>2</sub>O<sub>4</sub> and Yb doped ZnFe<sub>2</sub>O<sub>4</sub> magnetic ferrite by standard solid state method. The phase composition of the samples thus prepared was determined by X-ray diffraction (Shimadzu XRD-7000S diffractometer, CuK $\alpha$  radiation) and by analyzing energy dispersive X-ray spectra obtained on a JEOL JSM-5900L scanning electron microscope using an EDX detector. Lattice parameters were refined with FullProf-2010 software. The magnetic properties of the ytterbium doped zinc ferrite samples were studied in static magnetic fields of up to 30 kOe (2.4 MA/m) using a soft magnetic Hysteresis Graphs Meter (DX-2012SD).

### III. RESULTS AND DISCUSSION

X-ray diffraction characterization showed that, independent of the synthesis process, the major synthesis product was a Zn (Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> solid solution with a cubic spinel structure. In all instances, a single-phase material was only obtained at the rare earth content ( $x = 0.0125, 0.0250, 0.0375, 0.0500$ ) in Figure 1. Table 1 summarizes the structural characteristics of the solid solutions obtained. It follows from these data that the cubic cell parameter  $a$  of the rare earth- doped ferrites varies in a complex manner. The  $a$  cell parameter first increases and then, as a rule, decreases.

As seen in XRD spectra cubic spinel structure of zinc ferrite was found to be stable at final sintering temperature. The lattice constant and crystallite size of Zn(Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> calculated data are shown in Table 1. The lattice parameter obtained for pure zinc ferrite is in good agreement with the reported value.

The diffraction peaks corresponding to (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) reflection planes and the absence of any extra peak show that all the samples have attained single phase cubic structure. This implies that the Yb<sup>3+</sup> ions have been completely dissolved into the spinel lattice of zinc ferrite.

The lattice constant of ytterbium-substituted zinc ferrite is observed to be larger than that of zinc ferrite. An increase in lattice constant with increase in Yb<sup>3+</sup> ion content is expected because of the large ionic radius of Yb<sup>3+</sup> (0.858 Å) compare to that of Fe<sup>3+</sup> (0.67 Å).

The ytterbium substitution zinc ferrite changes in the lattice constant and this may probably be the reason for the observed shift of XRD peaks with ytterbium substitution. The theoretical (X-ray) density of zinc ferrite is in agreement with that of the zinc ferrite because it increases with increase in ytterbium concentration.

### A. Magnetic Hysteresis Loop Analysis

The magnetic hysteresis loops at room temperature were recorded by Magnetometer (DX-2012SD). The hysteresis loop data of Zn (Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> ( $x = 0.0000, 0.0125, 0.0250, 0.0375$  and  $0.0500$ ) are shown in Table 2 and Table 3. It is clear that by applying magnetic field of 10,000 Oe at room temperature, the saturation magnetization can be achieved for these samples. The value of  $M_s$  is found to be decreased as the amount of doping ions increased. It has been reported extensively that the smaller size particles exhibit the low value of saturation magnetization, which is due to their modified cationic distribution and surface disorder. According to the references, the magnetic moment of Zn<sup>2+</sup> is 0.3  $\mu_B$ , that of Yb<sup>2+</sup> is 0.85  $\mu_B$  and that of Fe<sup>2+</sup> is 4.9  $\mu_B$ -6.7  $\mu_B$ . Therefore, the value of  $M_s$  decreased as the amount of Yb doping ions with smaller magnetic moment was increased. According to brown's relation, the coercivity and saturation magnetization are given by the relation [3].

$$H_c = \frac{K_1}{\mu_0 M_s}$$

In equation, the  $K_1$  is magneto-crystalline anisotropy,  $\mu_0$  is vacuum permeability,  $M_s$  is saturation magnetization and  $H_c$  is coercivity. It has been observed that  $H_c$  and  $M_s$  of the prepared materials are inversely related by increasing substitution level and are in agreement with equation [3]. The value of  $H_c$  increased with the enhancement of doping ions of Yb<sup>3+</sup>. The low values of coercivity depicted the soft character of these materials which can be considered better candidate for switching, sensing and security applications. These low  $H_c$  values are necessary for electromagnetic (EM) materials. From the hysteresis loop, a number of primary magnetic properties of a ferrite material can be determined.

**Table1 Crystallite size of the ZnYbFe<sub>2</sub>O<sub>4</sub> samples at different temperatures**

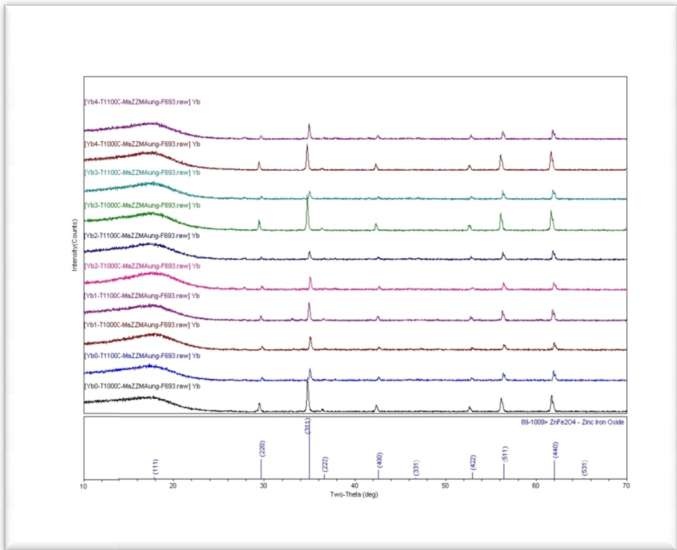
Sample (x)	Temperature 1000( °C)		Temperature 1100( °C)	
	Crystallite size (nm)	Lattice Constant (Å)	Crystallite size (nm)	Lattice Constant (Å)
x = 0.0000	44.02	8.47	42.46	8.47
x = 0.0125	36.78	8.47	43.80	8.50
x= 0.0250	44.29	8.47	44.76	8.49
x = 0.0375	41.18	8.48	47.30	8.49
x = 0.0500	49.91	8.49	51.77	8.55

**Table2 Magnetic hysteresis loop data for Zn(Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> at 1000°C**

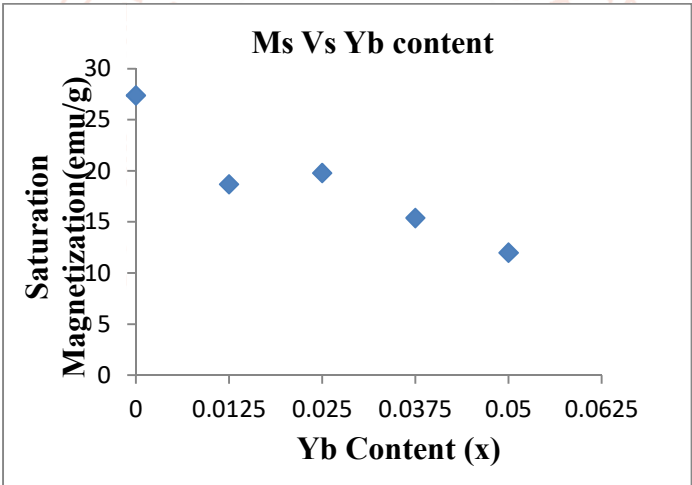
Composition of rare earth ions(x)	$M_s$ (emu/g)	$H_c$ (Oe)	$n_B$ ( $\mu_B$ )
0.0000	94.20	92.84	3.51
0.0125	76.5	119.26	2.83
0.0250	69.47	130.57	2.68
0.0375	67.47	143.77	2.61
0.0500	61.99	155.04	2.57

**Table3 Magnetic hysteresis loop data  
Zn(YbxFe1-x)2O4 at 1100°C**

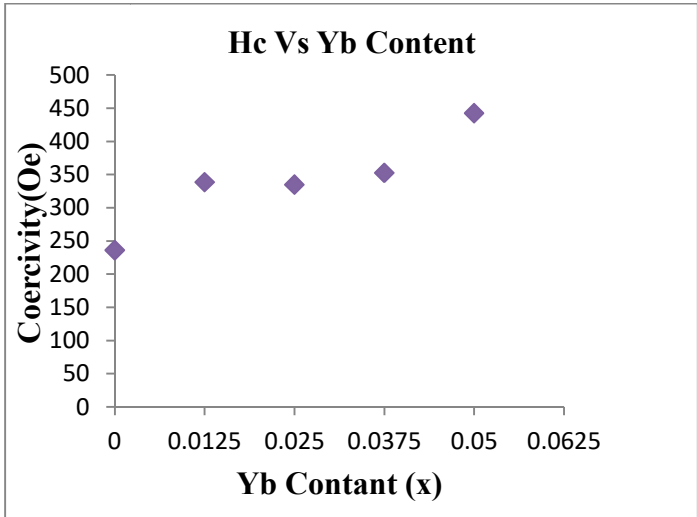
Composition of rare earth ions(x)	Ms (emu/g)	Hc (Oe)	n <sub>B</sub> (μ <sub>B</sub> )
0.0000	94.20	92.84	3.51
0.0125	76.5	119.26	2.83
0.0250	69.47	130.57	2.68
0.0375	67.47	143.77	2.61
0.0500	61.99	155.04	2.57



**Figure1 XRD pattern of the ZnYbFe<sub>2</sub>O<sub>4</sub> samples at different temperatures**



**Figure2 Variation of Saturation magnetization with different Yb Content (x) in Zn (Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> at 1000°C**



**Figure3 Variation of Coercivity with different Yb Content (x) in Zn (Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> at 1000°C**

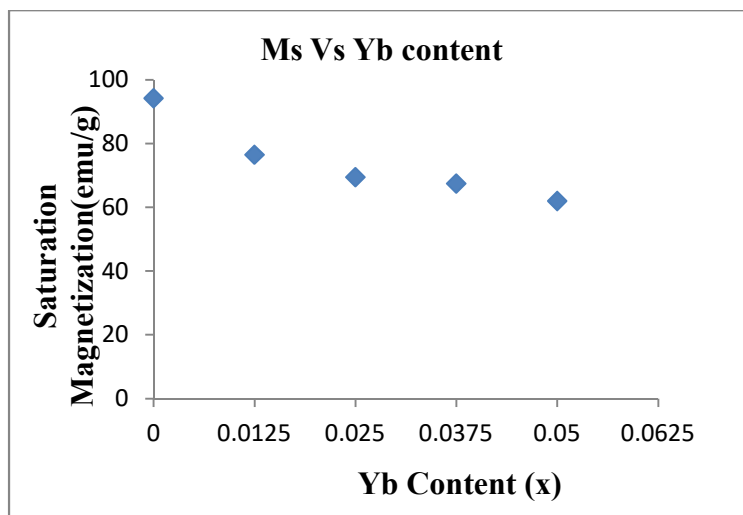


Figure4 Variation of Saturation magnetization with different Yb Content (x) in Zn (Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> at 1100°C

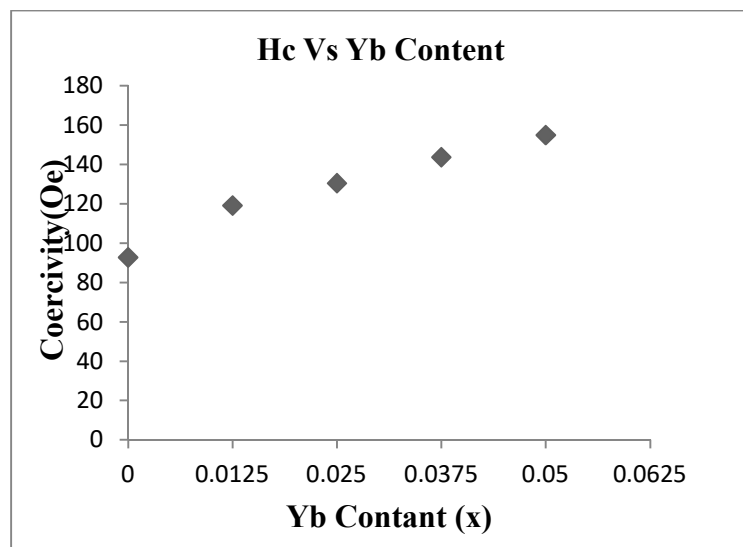


Figure5 Variation of Coercivity with different Yb Content (x) in Zn (Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> at 1100°C

### Conclusion

In this experiment of ytterbium doped on the zinc ferrite Zn (Yb<sub>x</sub>Fe<sub>1-x</sub>)<sub>2</sub>O<sub>4</sub> with x=0.0000, 0.0125, 0.0250, 0.0375 and 0.0500 were synthesized by using solid state method. XRD analysis confirmed the formation of single phase structure, without any secondary phase in all the compositions. The substitution of ytterbium in zinc ferrite has resulted in an increase in lattice constant. Moreover, it was observed that coercivity H<sub>c</sub> increased by increasing doping contents of Yb<sup>3+</sup> ions. It might be due to the effect of denser microstructure with Yb<sup>3+</sup> ions substitution in spinel lattice. This is proved that the substitution of small amount of ytterbium rare earth (RE) ions in ferrite can also tune the magnetic properties.

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